

OPTIONAL FORM 92 (7-90)

FAX TRANSMITTAL

of pages ▶

To	Paul Peronard	From	Julie Borgesi
Dept./Agency	EPA	Phone #	617-494-2434
Fax #	303-312-6962	Fax #	Volpe

NSN 7540-01-317-7388

5092-101

GENERAL SERVICES ADMINISTRATION

U. S. DOT/RSPA/Volpe Center
Environmental Preservation and Systems Modernization
DTS-30
55 Broadway, Kendall Square
Cambridge, MA 02142-1093

SDMS Document ID



2002588

Court,
Please pass along to
Parkers w/ appropriate
attachments.
Thanks!
Julie

DTS-30
FAX

Date: 6/18/02
Number of pages including cover sheet: 21

To:

Mel & Lerah Parker

Phone: 406-293-9705

Fax phone: 406-293-9705

CC: Paul Peronard

From:

Julie Borgesi

Phone: 617-494-2434

Fax phone: 617-494-2589

REMARKS:



Urgent



For your review



Reply ASAP



Please comment

Mel & Lerah,

Enclosed is our summary of why there were
differences between our lab results & yours.

Please provide us with a response or comments
by June 20 so that we may continue with
the restoration of your property without
delay.

Thank You! Julie



Memorandum

To: Peter Borowiec

From: Michael Oakland

Date: June 18, 2002

*Subject: Time Critical Asbestos Removal Project
Former Screening Plant Site
Differences in Agricultural Fill Test Data
Libby, Montana*

CDM has reviewed the testing procedures used by the property owner's testing laboratories, Sandberg Laboratories Ltd. and Norwest Labs to analyze stockpiled agricultural fill for use at the Screening Plant site. CDM retained Maxim Technologies, Inc. to perform an analysis of the stockpiled agricultural fill for compliance with the Government's specification. A second round of testing was performed by Maxim as a direct comparison to the tests performed by the property owner's testing laboratories. While all laboratories use a hydrometer to differentiate between the sand, silt and clay particles in the soil samples, fundamental differences in the methods used result in the variations in the results.

Maxim Technologies uses standard procedure ASTM D-422 that includes a combination of mechanical screening and hydrometer to determine the gradation of the entire sample with the hydrometer used to classify materials below the 0.075 mm size. The procedure is detailed and rigidly specified.

The property owner's laboratories, Sandberg and Norwest, used the less rigorous Bouyoucos or Modified Bouyoucos Methods. These methods use only a hydrometer after screening all soil at the 2 mm size. With the Volpe Center's authorization, we provided each laboratory with a copy of Maxim's test results and asked each laboratory to provide their interpretation of the differences. Their responses are attached.

The fundamental differences between the methods are that the ASTM procedure takes readings of the hydrometer at set times and then uses the time, temperature and reading data to compute an equivalent grain size while the Bouyoucos methods estimate when a certain grain size will have settled and takes a single reading at that time. However, pre-calculation of this time does not account for some aspects the hydrometer methodology such as the actual height of the hydrometer.

Mr. Peter Borowiec
June 18, 2002
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In addition, there also seems to be significant variability regarding what times should be used within the Bouyoucos method itself. Publications, such as the one attached to our previous memorandum, dated May 28, 2002, on the results for the Agricultural Fill testing, indicate a significant range of times being used by various laboratories which would affect the results. This is reflected in the comments on the differences made by the Sandberg lab.

In summary, after further reviewing the differences in the procedures, we continue to believe that the ASTM 422 method, as used by Maxim Technologies, is a more accurate procedure for determining grain size distribution and we believe that these results should be used in assessing the agricultural fill material for compliance with the Government's procurement specifications.

Attachments

Fax response by Sandberg
Fax response by Norwest

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FAX COVER SHEET

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TO:

Harold Jorndorfer

FAX NUMBER

1-617-452-8432

FROM:

Philip Sandberg

FAX NUMBER

1-403-320-1033

Harold To Mel Gordon -

The last time I talked with Mel we discussed the differences in the procedures used by the labs. The results will not be the same because of the differences. We discuss the sample to < 2.0m and over the 2 hour procedure, we could run the 24 hour test also, as that is the procedure the government sometimes requires. If you have other comments or observations please let me know?

*** HAVE A NICE DAY ***

Philip Sandberg

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**NORWEST
LABS**

Fax: (403) 327-8527

FACSIMILE TRANSMISSION

To: Harald Leiendecker	From: Norwest Labs Lethbridge
Name:	Name: Ken Mrazek
Company: CDM Federal Programs Corporation	Date: June 4 th , 2002
Fax No: 617-452-8432	3131 1 st Ave. S. Lethbridge, AB T1J 4H1

11 pages

Total number of pages transmitted including this sheet

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Harald,

Attached you will find a research publication that Norwest published in the Commun. Soil Sci. Plan Anal that relates to some of the issues with particle size by hydrometer. This research was done by our own scientists because of the inherent variability we were seeing between methods and modified methods. The method that Norwest used on these samples in question (Libby Asbestos Project) is the method as specified in our publication.

The technical notes from the Journal of Range Mgmt also identifies variability based on time, temp, and dispersion agent. These have all been addressed in the research that Norwest has done.

I hope this helps a bit in clarifying the differences between laboratories. Let me know if there is anything further that I can be of help for.

Ken Mrazek
Vice President
AgriFood Division
Norwest Labs.

<u>Our Locations</u>		
Edmonton	Calgary	Lethbridge
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COMMUN. SOIL SCI. PLANT ANAL. 32(5&6), 633-642 (2001)

STANDARD PROCEDURE IN THE HYDROMETER METHOD FOR PARTICLE SIZE ANALYSIS

John Ashworth, Doug Keyes, Rhonda Kirk,
and Robert Lessard

Norwest Labs, 9938-67 Avenue.
Edmonton, AB T6E 0P5, Canada

ABSTRACT

In a widely-used method for particle size analysis of soils, the weight percentages of sand, silt, and clay are calculated from the density of an aqueous soil suspension measured by hydrometer. There are many versions of the procedure, differing in the type of dispersing solution, the volume of the suspension, the time of settling before taking hydrometer readings, or in the method of correcting the raw readings. Our procedure avoids errors inherent in some versions of the method, which can cause discrepancies from expected values. The details of our procedure should interest those concerned with minimizing confidence limits in inter-laboratory surveys and with providing reliable particle-size distribution data to laboratory clients.

INTRODUCTION

A knowledge of particle size distribution is useful for characterizing soils in terms of their suitability for a range of agricultural, engineering, landscaping or reclamation purposes.

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The hydrometer method for particle size analysis of soils (1) is still in widespread use. It requires inexpensive equipment and only basic technical expertise. One version of the procedure can be used to construct soil particle size summation curves (2, 3, 4,), but we are concerned here with the original Bouyoucos (1) method for assessing weight percentages of the sand, silt and clay fractions.

In this procedure a density reading made 40 s after agitating a soil (<2 mm) suspension in a measuring cylinder is used for calculating % sand (defined as particles $>50 \mu$), and a later (2 h) reading is used to calculate % clay ($<2 \mu$). These particle size thresholds conform to the classification systems of both the United States Department of Agriculture and the Canadian Society of Soil Science. The % silt is the balance required for 100% of the sample weight.

Procedural details have altered over the many years during which this method has been in general use, possibly accounting for variations in results of inter-laboratory studies of particle size analysis using replicate samples [e.g., USU (5)]. There are many factors which can affect the results obtained.

Volume of Suspension

Bouyoucos (1, 6, 7) specified that, with the hydrometer inserted, a suspension with 50 g of soil was to be made up to a final level corresponding to 1,130 mL. Manufactured glass cylinders are available with this mark inscribed. The hydrometer (ASTM type 152H) displaces approximately 60 mL and so the volume of suspension is approximately 1,070 mL. However, the hydrometer reading increases as the volume is reduced, and other versions of the procedure specify a suspension volume of 1,000 mL [e.g., Sheldrick and Wang (8)].

Adjustments to Raw Hydrometer Readings

This step alters the estimated density by several grams L^{-1} . If it is not done correctly, large discrepancies in the calculated particle size distribution can be caused. Ways of adjusting for the density of a blank dispersing solution, and for laboratory temperature, are discussed below.

Time of Hydrometer Readings

The validity of Bouyoucos's (7) procedure was questioned by Gee and Bauder (9) on the grounds that the settling times which he had empirically adopted are inconsistent with the requirements of sedimentation theory. They found that % sand values calculated from a 40 s hydrometer reading often differed from

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weights retained on a 53 μ sieve, by more than 5% of the sample weight. They showed (Gee and Bauder, 10) that a reading made after 2 h leads to an estimate of % clay higher than the theoretical amount (2). These issues are also discussed below.

Cylinder Dimensions

Some documents (2, 4, 8) describe a measuring cylinder with the 1,000 mL mark near to 36 cm above the floor of the cylinder. Bouyoucos-type cylinders (with the engraved line corresponding to 1,130 mL) have an internal diameter near 62 mm, so that 1,000 mL reaches 33 cm above the floor, as is the case with the 1 L plastic graduated cylinders that we use. The effects of cylinder dimensions, which are minor, are discussed below.

Dispersing Agent

Heavy clay soils and soils contaminated with sodium salts may not disperse completely if insufficient dispersing chemical is used. In terms of efficacy, Bouyoucos (7) said there is little to choose between sodium pyrophosphate and metaphosphate. The final concentration of reagent determines the density and viscosity of the solution.

In view of all the above possible sources of discrepancy, there is a clear need for a unified, validated procedure.

MATERIALS AND METHODS

In our procedure, a stock dispersing solution is prepared weekly from tetrasodium pyrophosphate decahydrate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$, 500 g) made up to 10 L in deionized water. Soils are dried in a current of warm air (45°C) and pulverized to pass a 2-mm sieve. A subsample (50 g) is treated in a 1 L plastic cup with 100 mL of the stock solution. The mixture is made up to about 250 mL and left overnight (16 h). It is then transferred to a metal cup with indentations (milk-shake cup), mixed with a high speed blender for 5 minutes and rinsed into a measuring cylinder. The suspension is made up exactly to the 1,000 mL mark with deionized water from a large stock at laboratory temperature. The temperature of the suspensions is not controlled, but it never drifts more than 2°C at most in the course of the day.

Homogenizing the suspension by repeatedly inverting the capped cylinder (4, 7) may be impractical when many samples are processed daily. An alternative is to rake the suspension from top to bottom with a plunger (2). In cursory tests, we found that this method was as effective as end-over-end mixing. After mixing

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by either method, a standard ASTM type 152H hydrometer (9) is carefully inserted 20 s ahead of the reading time. The stem scale is read at the top of the meniscus. The stem of the hydrometer is kept free of grease so that a meniscus forms properly. We estimate the 40 s reading to the nearest 0.5 g L^{-1} and the later, far more stable reading (6 h after mixing) to the nearest 0.1 g L^{-1} under good light with the aid of a magnifying glass.

The above procedure was verified using a batch of 22 soils provided by the University of Alberta. These surface and sub-surface materials, taken from various locations in Alberta, varied widely in particle size distribution, organic matter (2 to 10%) and lime content (0 to 25%).

Verifying the 40 s Sand Settling Time

Subsamples (50 g) of dried soil ($<2 \text{ mm}$) were prepared and analyzed by the procedure given above. Replicate subsamples (50 g) were also wet-sieved through 53μ (270 mesh) and the dry weight of retained sand was measured, thus obtaining also the weight of silt and clay passing the sieve.

Verifying the Hydrometer Reading

Suspensions of fine silt and clay were obtained by allowing suspensions of whole soil, prepared as described, to settle for about 0.2 h in order to separate all sand and coarser silt. The upper portion, about 600 mL, was then removed by siphoning. It was made up again to 1 L with 0.5% sodium pyrophosphate decahydrate solution. The suspension was raked with the plunger and the hydrometer inserted. Readings were stable for several minutes and were estimated to the nearest 0.1 g L^{-1} . These suspensions were finally transferred to a pre-weighed pan and dried to constant weight in a current of air at 45°C , in order to obtain the weight of suspended solids. A correction was made to these weights for the dry weight of $\text{Na}_4\text{P}_2\text{O}_7$ (mean 3.1 g) in a 1 L blank suspension.

Temperature-Dependence of Hydrometer Readings

The above density measurements were made in the range $22\text{--}24^\circ\text{C}$, as indicated by a mercury in glass thermometer calibrated against an NBS standard. Before being dried, the suspensions of fine silt and clay were left overnight in a separate room maintained near 15.0°C . Each was then thoroughly agitated and homogenized using the plunger; the hydrometer readings were repeated, and the actual suspension temperature measured.

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RESULTS AND DISCUSSION

Correcting the Raw Hydrometer Reading

Two approaches have previously been used in correcting the raw hydrometer reading (R). One way is to subtract an actual blank reading (R_L) made in the dispersing solution (with no soil) at the same temperature as that of the soil suspensions (8, 9). The other way is to subtract a fixed, standard blank reading, whose value corresponds to a standard temperature, and then to make a correction for the difference between that standard and the actual temperature (7).

Using the first approach, we found a direct linear relationship (with $r^2=0.998$) between the weights of fine silt and clay in 1000 mL suspensions, and blank-adjusted hydrometer readings taken at 22–24°C soon after homogenizing them (Figure 1). This finding supports the view that blank-adjusted readings indicate with sufficient accuracy the density D (in g L^{-1}) of suspended solids (8, 9), according to the equation:

$$D = R - R_L \quad (1)$$

Judging from hydrometer readings made in the 1000 mL suspensions and in dispersing solution, at various temperatures in the range 13–24°C, the average value of dR/dT (approximately $0.3 \text{ g L}^{-1} \text{ }^\circ\text{C}^{-1}$) scarcely differs from dR_L/dT (approximately $0.4 \text{ g L}^{-1} \text{ }^\circ\text{C}^{-1}$). Therefore, dD/dT is very small. Having subtracted an actual blank reading R_L , further correction, to a standard temperature within several °C of the actual laboratory temperature, would have little effect on D .

The alternative of correcting to a standard 20°C and subtracting a fixed blank reading (7) performs the same function as subtracting R_L . It avoids the need for making an actual R_L , with its associated blank reading error. However, it unfortunately also introduces the risk (11) of inadvertently subtracting an actual R_L which, depending on the laboratory temperature, can differ by several g L^{-1} from the true standard blank reading appropriate to 20°C.

We therefore favor using equation [1]. The practice of subtracting an actual R_L from R also offsets any discrepancies among different hydrometers in the positioning of the scale in the stem during manufacture.

Time of Clay Reading

Equation [1] can be used to estimate the proportion of any particle size fraction, if the hydrometer is read after the appropriate settling time. In Gee and Bauder's (9) equations [13] and [14] (based on the theory of Day (2)), setting the difference between particle and solution densities at 1.6 g mL^{-1} , and using $0.95 \times 10^{-3} \text{ kg m}^{-1} \text{ s}^{-1}$ as the solution viscosity at 22–24°C (by interpolation

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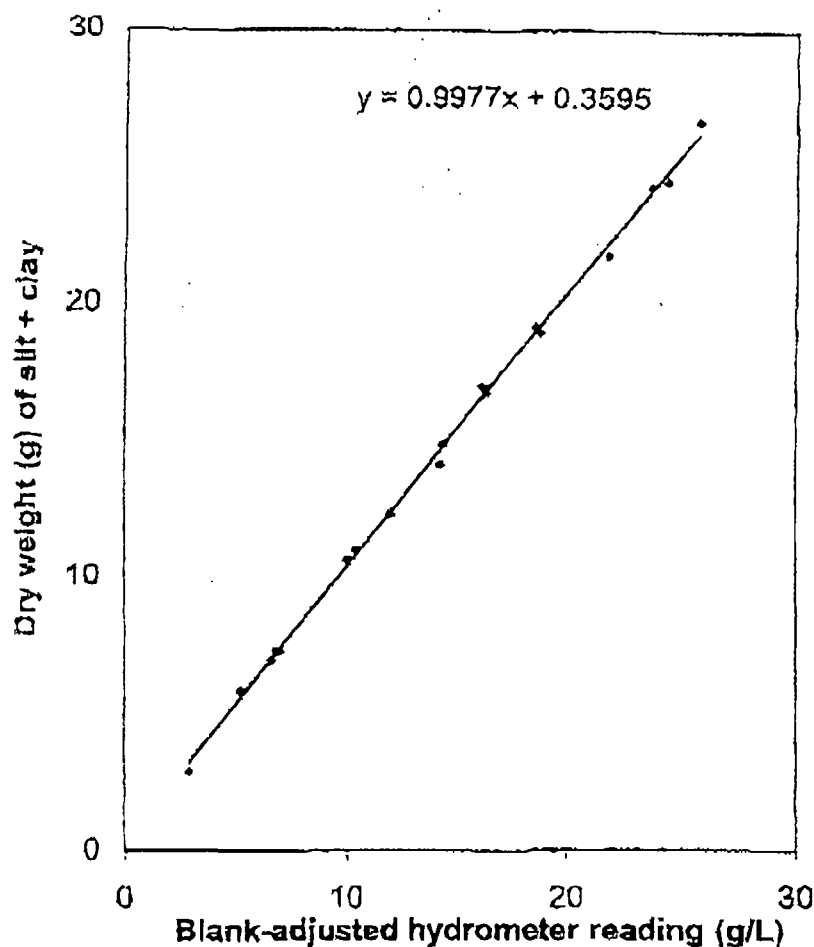


Figure 1. Weight of suspended solids versus blank-adjusted reading.

from their Table 15-1), a 6 h settling time in the cylinders that we use corresponds to an equivalent particle size diameter of $2.0 \pm 0.1 \mu$, for hydrometer readings in the range $10\text{--}30 \text{ g L}^{-1}$.

Using a 6 h reading (R_{6h}) therefore removes the high bias in % clay associated with a 2 h reading, noted by Gee and Bauder (10), while still allowing preparation and readings to be done in a regular working day by the same technician.

Cylinder Dimensions

The effective hydrometer settling depth is insensitive to the distance from the surface to the floor of the cylinder, and is scarcely affected by small changes to the cross-sectional area of the cylinder [using equation [10] of Gee and Bauder (9)].

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Sand Reading

We also found a linear relationship for the batch of 22 soils (Figure 2) between weights of silt and clay particles passing 53μ , and values calculated using equation [1] from blank-adjusted 40 s hydrometer readings (R_{40s}) in 1000 mL suspensions at 23°C . The near 1:1 relationship obtained (with $r^2=0.989$) supports the empirical choice of 40 s for the sand reading time (1).

Calculations

The following equations are therefore considered suitable:

$$\% \text{ clay} = (100/w) (R_{60} - R_L) \quad [2]$$

in which w is the weight of dry soil in 1000 mL of suspension;

$$\% \text{ sand} = 100 - (100/w) (R_{40s} - R_L) \quad [3]$$

and

$$\% \text{ silt} = 100 - \% \text{ sand} - \% \text{ clay} \quad [4]$$

The blank readings R_L in equations [2] and [3] are actual readings, which will not have the same value if the suspension temperature drifts.

Calcium Carbonate and Organic Matter

Many workers [e.g., Sheldrick and Wang (8)] have pointed out that lime and organic matter must first be removed, if a true assessment of size fractions of mineral particles is required. Even though these components had not been taken out of the soils we tested, there were no seriously outlying points in Figures 1 and 2. Suspended lime and organic matter fractions seem in effect to have conformed to an equivalent size distribution of mineral particles. Bouyoucos (6, 7) suggested that particle size distribution may for practical purposes be characterized by analysis done on whole soil and that, if desired, lime and organic matter can be quantified separately.

Hydrometer Consistency

The possibility that individual standard ASTM type 152H hydrometers might vary significantly in their density readings R is not explicitly covered in the literature cited below, and needed to be considered. To check variability, we tested

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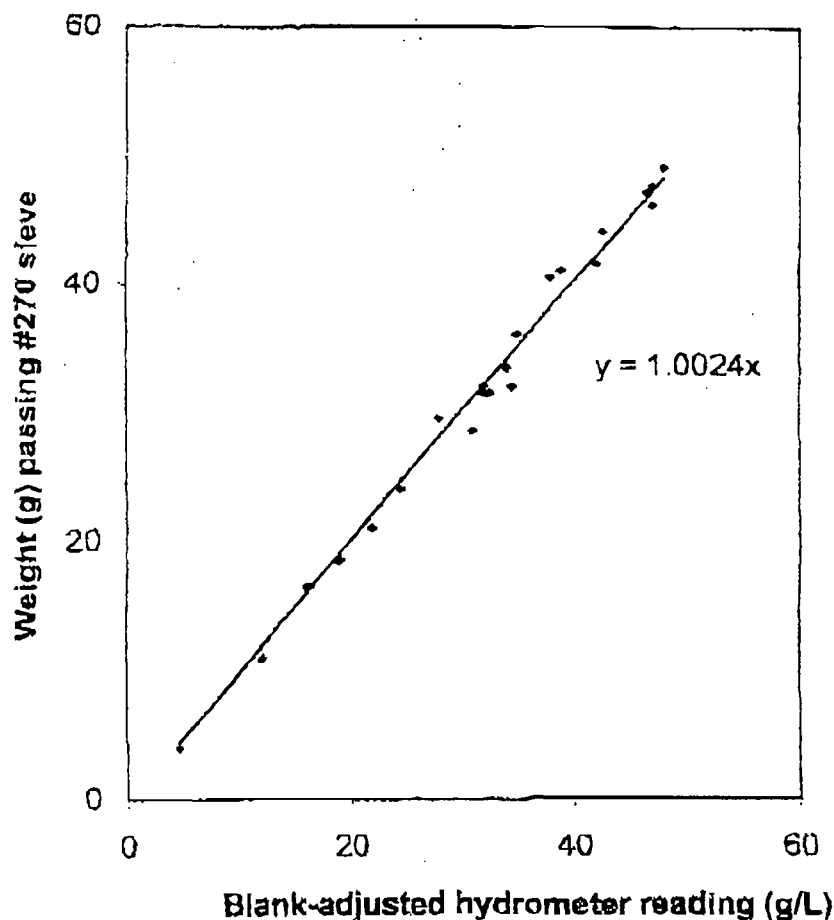


Figure 2. Sieve analysis versus blank-adjusted readings (40 s).

salt solutions of known density, using ASTM type 152H hydrometers already in use in our laboratory and new ones purchased from different suppliers. The results indicated that blank-adjusted readings made with hydrometers of this type generally agree within $\pm 1 \text{ g L}^{-1}$. Equations [1] through [4] can therefore be expected to apply for all standard hydrometers of this type.

Sample Preparation

Sample preparation procedures could be a further source of inter-laboratory discrepancy. Ball mills or other grinders which could break sand particles into smaller fragments should be avoided. The risk of damaging soil particles is considered low with the flail-type pulverizing equipment we use, though we have not investigated this possibility. The advantage of analyzing subsamples of dried, pul-

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verized material lies in avoiding both the difficulty of preparing representative subsamples of moist, as-received soils and the need for a separate moisture content determination on another such subsample (2, 8).

CONCLUSIONS

For the soils we studied, a blank-corrected hydrometer reading taken 40 s after mixing a suspension of 50 g soil in exactly 1 L of solution gave an accurate estimate of the weight percentage of material passing a 53 μ sieve, and hence of sand content.

The clay content was calculated from a blank-adjusted 6 h reading, thus eliminating the high bias in results obtained with a 2 h reading (10), while still allowing the sand and the clay readings both to be made by one technician conveniently in the course of a working day. To guard against variability among hydrometers and in readings made by different technicians, it is advisable for one technician to measure R and R_c , using the same hydrometer.

Over a wide range in the temperature of measurement, blank-adjusted readings were in good agreement with dry weights of suspended solids. Having subtracted an actual blank reading from suspension densities measured at nearly the same temperature, an additional temperature-standardization of the adjusted reading is not required.

The validity of equations [2] and [3] is unlikely to be affected by small differences in design of measuring cylinders, or in the amount or type of dispersing chemical used. However, errors in estimates of particle fractions of the order of 5–10% of sample weight can be caused by using suspension volumes other than 1,000 mL, by using inappropriate reading times, or applying a temperature-correction to the hydrometer reading in addition to an actual blank adjustment.

ACKNOWLEDGMENTS

We thank Rob Ellis for querying the need for a temperature correction to blank-adjusted hydrometer readings. Jim Robertson and Don Pluth of the University of Alberta provided the soils and helpful discussion. Cathy Ngo, Martine Longpré, and Alistair Matthew provided technical assistance.

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Memorandum

To: Peter Borowiec

From: Harald Leindecke
Michael Oakland

Date: May 28, 2002

Subject: Grain Size Distributions
Libby Asbestos Project

This memorandum summarizes differences in the results of grain size distribution testing conducted on samples of agricultural fill from the Screening Plant site in Libby, MT. Samples were tested by the project's laboratory (Maxim Technologies, Inc.) and laboratories (Sandberg & Norwest) retained by the property owners, Mel and Lerah Parker. Maxim was retained by CDM to conduct laboratory testing of samples collected from a stockpile of Agricultural fill for potential use as backfill material on the Parker's property. Duplicate samples were collected by the Parkers that were tested by Sandberg Laboratories Ltd and Norwest Laboratories. The results of the testing completed at the laboratories retained by the property owners differed significantly from those tested by CDM as shown in Table 1 for the Sandberg lab and in Table 2 for the Norwest lab. In addition, the results between the two laboratories retained by the property owners also differed significantly.

Table 1: CDM vs. Sandberg Test Results

Sample	Particle-Fractions		
	Sand	Silt	Clay
1R-13752* / 15**	25.9/16.2 (+ 9.7%)	58.1/ 61.4 (- 3.3 %)	16.0/ 22.4 (-6.4 %)
1R-13753 / 16	25.6/ 16.2 (+ 9.4%)	59.3/ 61.4 (- 2.1 %)	15.1/22.4 (-7.3 %)
1R-13754 / 17	25.3/19.8 (+ 5.5 %)	60.6/ 56.0 (+ 4.6 %)	14.1/24.2 (-10.1%)
1R-13755 / 18	26.3/16.2 (+ 10.1%)	60.6/ 59.4 (+ 1.2%)	13.1/24.4 (-11.3 %)

Peter Borowiec

May 28, 2002

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1R-13756 / 19	25.7/16.0 (+9.7 %)	61.3/60.6 (+ 0.7%)	13.0/23.4 (-10.4 %)
1R-13757 / 20	31.1/28.0 (+3.1%)	55.9/52.0 (+ 3.9%)	13.0/20.0 (-7.0 %)
1R-13758 / 21	29.2/20.0 (+9.2 %)	58.8/57.2 (+ 1.6%)	12.0/22.8 (-10.8 %)
Average Difference	+8.1 %	+2.5 %	-9.0 %

*Italics: Maxim/CDM

**Bold: Sandberg Laboratories Ltd/Parker

Table 2: CDM vs. Norwest Test Results

Sample	Particle-Fractions		
	Sand	Silt	Clay
1R-13753 / 16	25.6/ 15.3 (+ 10.3%)	59.3/66.6 (- 7.3 %)	15.1/17.6 (-2.5 %)
1R-13755 / 18	26.3/11.0 (+ 15.3%)	60.6/70.0 (-9.4 %)	13.1/19.0 (-5.9 %)
1R-13757 / 20	31.1/18.6 (+12.5%)	55.9/ 64.8 (-8.9%)	13.0/ 16.6 (-3.6 %)
Average Difference	+12.7 %	-8.5 %	-4.0 %

*Italics: Maxim/CDM

**Bold: Norwest Laboratories/Parker

The CDM laboratory results indicate a higher percentage of sand while the property owner's laboratories indicate a higher percentage of clay. The silt fraction varied between the comparisons with the Sandberg laboratory indicating less silt than the CDM lab while the Norwest laboratory results indicated more silt than the CDM lab.

Maxim's tests were conducted in accordance with standard procedure ASTM D-422 which uses sieves for particle sizes larger than 75 μm a hydrometer for smaller sized particles. The property owner's laboratories, Sandberg Laboratories Ltd and Norwest Laboratories conducted their testing using an Improved Bouyoucos Hydrometer method which uses a hydrometer only. The hydrometer testing procedures used in either of the two approaches are also different with the ASTM procedure having a longer duration and taking more readings than the Improved Bouyoucos method.

ASTM methods are generally used as the standard for engineering projects. While we have not conducted a specific comparison of the two methods of determining grain size, published material of others who have, generally warn that the Improved Bouyoucos method is not accurate and tends to over-estimate the clay content which is consistent with

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the comparison of results on our project. A technical note discussing the comparison is attached.

In summary, we believe that inconsistent testing methods from those specified by us for this project are the principal reason for the difference in results. Following our brief review of the method and the published literature, we would conclude that the ASTM procedure is more accurate.

Attachment: Technical Note: Comparison of hydrometer settling times in soil particle size analysis by Carolyn C. Bohn and Karl Gebhardt, Journal of Range Management 42(1), January 1989.

Technical Notes

Comparison of hydrometer settling times in soil particle size analysis

CAROLYN C. BOHN AND KARL GEBHARDT

Abstract

Although soil texture is important to plant growth, cultivation, hydraulic conductivity, and soil strength, laboratory procedures for determining particle size distribution can be confusing. A number of settling times have been proposed for the hydrometer method used to analyze the fine earth fraction of soils. To separate sand and silt, hydrometer readings at 30 and 60 seconds, 35 seconds, or at 40 seconds have been recommended. To distinguish between silt and clay, recommendations have been made for readings at 6–8 hours and 12–15 hours, 1.5 and 24 hours, 2 and 24 hours or at 8 hours. In this study, no significant differences in estimates of sand content were found between readings made at 30 and 60 seconds and at 40 seconds. However, estimates from readings on both sides of the silt-clay separation (at 6 hours and 12 hours) showed a significant variation of clay content within the sample probably due to an inadequate method of splitting the soil samples into subsamples. Clay estimates from 2-hours readings differed significantly from the average estimate of the split sample 6/12-hours readings. Numerical differences were seen among particle size estimates from various methods; if the soil texture is near a division between 2 classes, these differences may result in different textures being assigned.

Key Words: particle size distribution analysis, soil texture, soil analysis methods

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Research was funded by the United States Department of the Interior, Bureau of Land Management, Saval Ranch Research and Evaluation Project, University of Nevada Agricultural Experiment Station Project 665. The authors thank Shirley Hudson for statistical advice.

Manuscript accepted 21 June 1988.

Soil scientists have long recognized the importance of soil texture to plant growth, cultivation, hydraulic conductivity and soil strength. However, managers preparing to determine soil texture are sometimes confused by conflicting procedural recommendations. Textural classes can be determined either in the field or by a laboratory particle size distribution analysis. The laboratory analysis most often available to land managers is some version of the hydrometer method first introduced by Bouyoucos (1927). Day (1965) and the American Society for Testing and Materials (A.S.T.M.) (1972) have detailed the procedure generally accepted by land management agencies and an updated edition was recently released (Gee and Bauder 1986). In this method, a measured amount of soil is suspended in water and the suspension density is determined with a specialized hydrometer. As soil particles settle, the suspension density decreases. Because larger particles settle faster, the particle size and summation percent remaining for that size can be calculated for each measurement time using the observed hydrometer level, and these results are graphed. From the relationship on the graph, the percent of a particular particle size class can be estimated (Day 1965).

The accuracy of the size class distribution estimate depends on a constant temperature, careful particle dispersal, and proper timing of the density observations. In the past, chemical dispersal has often been accomplished with a 5% Calgon (water softener trade name) solution. However, the preparation currently on the market may not contain phosphates, which are necessary for good dispersal; the label should be examined and a dispersing agent such as sodium hexametaphosphate purchased from a chemical supplier, if necessary. Phosphates are particularly important for soils high in

to ex organic matter. While Day (1965) recommends soaking at least 10 minutes, the A.S.T.M. (1972) recommends several hours and the most recent laboratory procedures now specify soaking overnight (Gee and Bauder 1986). Likewise, the practice of using milkshake mixers for mechanical particle dispersal has been questioned (Doolittle 1957, Walker and Hultine 1973), but continues in many labs. It is generally thought that a reciprocal shaker is slow and gentle but may not break up cemented soils, while the mixer is fast and vigorous but may break down sands affected by weathering. Some of the problem may be avoided by making plastic mixing paddles. Regardless of what methods are selected for particle dispersal, the exact procedure should be described and used consistently.

The timing of the hydrometer readings depends on the size classification system being used. The separation between silt and clay used by the U.S.D.A. (0.002 mm), usually occurs between 8 to 12 hours of undisturbed settling, but the exact time cannot be identified beforehand. Measurements of the solution density at 6-8 hours and again at 12-15 hours will yield data which brackets the actual time at which all the silt size particles have settled and therefore brackets the corresponding percent clay on the particle size summation percentage graph. Similarly, the break between sand and silt (0.05 mm) occurs between 30 to 60 seconds of settling time and can be estimated from a graph showing these data points.

In order to reduce laboratory time, researchers have proposed a variety of observation times which approximate the desired particle size and depend on extrapolation rather than surrounding the size with measurements. Bouyoucos (1962) suggested that readings at 40 seconds and 2 hours were sufficient, but Gee and Bauder (1979) stated that 2 hours over-estimated the clay fraction and developed a method of weighted averages using 1.5 and 24 hours readings for the clay fraction. Patrick (1958) used 35 seconds and 8 hours. The methodology endorsed by the A.S.T.M., and generally accepted by land management agencies, uses readings at 30 seconds, 3, 10, and 30 minutes, and 1.5, 4.5, and 12 hours (Day 1965). However, A.S.T.M. (1972) also recommended 2, 5, 15 and 30 minutes, and 1, 4, and 24 hours, and the 2nd edition of the standard methods manual (Gee and Bauder 1986) cites 30 and 60 seconds, 3, 10, and 30 minutes and 1, 1.5, 2, and 24 hours. To save lab time and expedite the analysis, some labs terminate the analysis at 6-8 hours. Although the best estimates would be expected from density observations which bracket the breakoff between size groups, in some cases, estimates from shorter settling times may suffice. It is important to identify what effect shorter settling times may have on soil texture estimations. We report here results from observations made at 30, 40, and 60 seconds and 2, 6, and 12 hours.

Methods

Twelve soil samples were collected from a 1,200 foot reach of streambank in the Independence Mountains, 50 miles north of Elko, Nevada. As is typically true, these riparian soils have not been classified to series, but are generally permeable alluvium. No effort was made to select a wide range of textures. A vertical cut was made into the streambank at each sample site and the samples were collected from the freshly exposed soils. The samples were oven-dried and sieved and the fraction passing the 2-mm sieve saved for analysis by the hydrometer method (Day 1965). Two subsamples from each sample were derived by gently inverting the original sample several times in a closed container and spooning out 40 grams for each subsample. Each subsample was then uniformly dispersed by soaking in 5% Calgon solution for 12 minutes followed by stirring for 5 minutes in a milkshake mixer. Each subsample was poured into a Bouyoucos tube, diluted with enough distilled water to bring the suspensions to 1,000 ml., and mixed with a plunger. The suspension density was read with an A.S.T.M.

152 H Soil Hydrometer at 30, 40, and 60 seconds, 2, 6, and 12 hours. Thus, data were collected to test 3 methods: (1) 30 and 60 seconds to sample either side of the sand/silt division, and 6 and 12 hours to sample either side of the silt/clay division (the "bracketing" method); (2) 40 seconds and 6 hours to estimate both sand and clay separation points; (3) 40 seconds and 2 hours to estimate both sand and clay separation points. All readings were adjusted for temperature (Day 1965) and particle size diameters vs. summation percentages were graphed for each subsample to determine percentages for specific particle size groups.

The resulting sand and clay percentages were analyzed separately. Using the 12 samples as replications, paired T-tests ($n=12$) were employed to test the sand estimates between: (1) the 2 subsamples each using the 30/60 seconds bracket method; (2) 30/60 seconds bracket vs. 40 seconds and 6 hours method; (3) 30/60 seconds bracket vs. the 40 seconds and 2 hours method; (4) average of the two 30/60 seconds bracket subsamples vs. 40 seconds and 6 hours; (5) average of the two 30/60 seconds brackets vs. 40 seconds and 2 hours. Similarly, paired T-tests were employed to test the clay estimates between: (1) the 2 subsamples each using the 6/12 hours bracket method; (2) 6/12 hours bracket method vs. the 6 hours and 40 seconds method; (3) 6/12 hours bracket vs. 2 hours and 40 seconds method; (4) average of the two 6/12 hours bracket subsamples vs. 6 hours and 40 seconds; (5) average of the two 6/12 hours brackets vs. 2 hours and 40 seconds method.

Results

The percentages of particle size classes are displayed in Tables 1 and 2. Percent sand estimates showed no significant differences

Table 1. Percent sand estimates.

30:60 Seconds Bracketing Method					
	Sub-sample 1	Sub-sample 2	Average of sub-samples	40 Sec. & 6 hours	40 Sec. & 2 hours
Sample 1	51.1	52.0	51.8	50.0	50.0
Sample 2	54.0	54.5	54.3	52.5	52.5
Sample 3	50.5	47.5	49.0	49.5	49.5
Sample 4	48.0	49.5	48.8	50.0	50.0
Sample 5	41.0	46.5	43.8	44.5	44.5
Sample 6	44.0	47.0	45.5	47.0	47.0
Sample 7	50.0	53.0	52.5	55.0	55.0
Sample 8	57.0	59.5	58.3	59.5	59.5
Sample 9	44.0	44.5	44.3	46.5	46.5
Sample 10	73.0	73.0	76.5	76.5	76.5
Sample 11	45.0	41.0	43.0	42.0	42.0
Sample 12	43.5	44.0	43.8	44.0	44.0

between any groups (Table 3). Because the sand/silt separation occurs within a very small time range, any reading between 30 and 60 seconds should be reasonably close to the actual break. Analysis of the clay groups showed a significant difference between the 2 sub-samples which are both estimated by the standard 6/12 hours bracket method. This was apparently due to variability within the sample and the difficulty of adequately mixing and splitting soil samples into comparable subsamples. For these samples, the difference in clay estimates between the standard 6/12 hours bracket method and the 6 hour method does not appear to be greater than the variability encountered in splitting the sample by hand. However, the 2 hours clay estimate differed significantly (.01 level) from the average of the two 6/12 hours clay estimates, and from the unaveraged 6/12 hours estimate at a lesser level of significance (.10). There were some noticeable numerical differences between the estimates from the 6/12 hours bracket method and both methods using shorter settling times, probably due to the distance

Table 1. Percent clay estimates.

6/12 Seconds Bracketing Method					
	Sub-sample 1	Sub-sample 2	Average of sub-samples	40 Sec. & 6 hours	40 Sec. & 2 hours
Sample 1	8.5	6.0	7.3	2.5	2.0
Sample 2	10.5	7.5	9.0	6.0	3.0
Sample 3	8.5	7.5	8.0	5.5	11.5
Sample 4	10.5	5.0	7.8	2.5	2.0
Sample 5	13.0	9.0	11.0	7.0	7.0
Sample 6	12.0	9.0	10.5	7.5	4.5
Sample 7	12.0	12.0	12.0	14.0	14.5
Sample 8	10.5	9.0	9.8	19.0	6.0
Sample 9	13.0	15.5	16.8	16.5	15.0
Sample 10	6.0	6.0	6.0	6.0	5.0
Sample 11	8.0	8.0	8.0	7.5	2.5
Sample 12	10.5	9.5	10.0	7.5	7.0

Table 3. T-values from paired comparisons of % sand and % clay estimates by different methods ($\alpha = 1\%$).

Particle size	Methods compared	T-value
Sand	30/60 sec bracket vs. 30/60 sec	1.5844
Sand	30/60 sec bracket vs. 40 sec & 6 hours	0.1978
Sand	Average bracketing vs. 40 sec & 6 hours	1.1010
Sand	30/60 sec bracket vs. 40 sec & 2 hours	0.3876
Sand	Average bracketing vs. 40 sec & 2 hours	1.1010
Clay	6/12 hour bracket vs. 6/12 hour bracket	4.6000**
Clay	6/12 hour bracket vs. 40 sec & 6 hours	0.2025
Clay	Average bracketing vs. 40 sec & 6 hours	1.8814
Clay	6/12 hour bracket vs. 40 sec & 2 hours	2.1454*
Clay	Average bracketing vs. 40 sec & 2 hours	3.2035**

**Significant difference at .01 level

*Significant difference at .10 level

of extrapolation over the graph. Because readings at 6 and 12 hours bracket the actual silt/clay break, that method, in theory, provides the more accurate estimates. Where the textural classification is borderline, even small numerical differences may change the class; this occurred with 2 samples.

Conclusions

Hydrometer readings anywhere between 30 and 60 seconds should reasonably estimate the percent sand in a soil sample. However, the percent sand estimate generally is not a problem for scientists wanting to save lab time. The silt/clay separation requires several hours of settling time, although opinions vary on the number of hours needed. Because there was no statistical difference between the 6/12 hours method which bracketed the silt/clay separation and the method which terminated at 6 hours, 6 hours of settling should be adequate, at least for some soils. However, the numerical differences observed between the 2 methods could occasionally define different textural classes, and the longer method theoretically provides the greatest accuracy. Care should be taken to thoroughly mix dry samples. The level of precision required will depend on the intended use of the data.

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